Fermilab

Particle Physics Division
Mechanical Department Engineering Note

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Project Internal Reference:

Project: SNAP

Title: SELECTION OF AN EPOXY TO BOND SILICON TO ALUMINUM NITRIDE

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Reviewer(s):

Key Words: SNAP, ADHESIVE, SILICON

Abstract Summary:
Material Testing of three epoxies is reported in MD-ENG-103. Selection criteria of an epoxy for bonding Silicon to Aluminum Nitride with an operating temperature near 150K is given. The criteria is calculated for each epoxy as a function of temperature.

Applicable Codes:
N/A

Introduction:

An epoxy needs to be selected for bonding Silicon to Aluminum Nitride with an operating temperature of ~150K. Epoxies generally have a Coefficient of Thermal expansion that is roughly an order of magnitude greater than the substrate material. The large thermal expansion difference between the substrate and the epoxy creates a stress in the bond material. If the stress is greater than the epoxy strength, the joint fails. A criteria is presented for selecting an appropriate epoxy based on the cryogenic properties for Tracon-F113, Epotek 301-2, and Hysol 9361 epoxies as reported in MD-ENG-102.

Summary Tables from MD-END-103:

Table 1. Modulus in KSI.

Epoxy Name	295K	250K	200K	150K	100K
Tra-Con F113	357	519	616	886	1,106
Epotek 301-302	531	596	649	833	1,014
Hysol 9361	155	239	561	823	1,132

Table 2. Maximum Stress Tested (not yield strength) in PSI.

Epoxy Name	295K	250K	200K	150K	100K
Tra-Con F113	4,798	3,527	4,272	5,982	7,737
Epotek 301-302	9,664	4,125	4,471	5,681	6,783
Hysol 9361	2,400	1,736	3,912	4,641	4,225

The stress listed for 295K is ultimate strength taken from the Fermi Tensile Data. The remainder of the data is taken from the PMIC report. PMIC reported the stress on the sample at the maximum strain measured. In most cases, the test ended at 0.68% strain. The Hysol samples were tested at a much lower strain due to sample failures at the warm ends f the dogbone.

Table 3. dL/L

Epoxy Name	295.5 K	250K	200K	150K	100K
Tra-Con F113	0	-.00339	-.00693	-.00889	-.01084
Epotek 301-302	0	-.00284	-.00555	-.00778	-.00975
Hysol 9361	0	-.0047	-.00869	-.01133	-.01335

A Guideline for Understanding if the Epoxy will Fail Due to Thermal Stresses:

Modulus = Stress / strain
Strain = dL/L
dL/L(epoxy) = CTE (epoxy) * dTemp

Combining to get thermal stress in the bonded joint, and assuming CTE of the substrate is zero compared to the epoxy.

Stress = Modulus * CTE(glue) * dTemp

Assign a criteria that the stress must be below 1/3 yield strength of the glue

1/2 > Modulus (glue) *CTE (glue) *dTemp / yield stress (glue)

Applying the Guideline to the Data Collected:

The stress reported in the data sets is not the epoxy yield strength. However a conservative guideline can still be determined if the maximum stress achieved is used in the calculations as a proof stress. The proof stress used in the formula was the higher of the room temperature ultimate strength or the maximum stress applied to the sample at temperature. Since the strength is known to increase as the temperature decreases, this is a conservative approach.

Table 4. Modulus (glue) *CTE (glue) *dTemp / yield stress (glue)

Epoxy Name	295.5 K	250K	200K	150K	100K
Tra-Con F113	0	0.36	0.89	1.32	1.55
Epotek 301-302	0	0.17	0.37	0.67	1.0
Hysol 9361	0	0.47	1.2	2.0	3.5

Epotek 301-2 has the lowest bond stress. Although the epoxy does not meet the less than 0.5 criteria at temperatures below 200K, it does not necessarily mean that the joint will fail. The number for yield stress is less than the actual expected epoxy yield strength.

The Tensile data collected and reported for the epoxy material properties was taken at high pull rates. Epoxy, as with most plastics, is a visco-elastic material which responds differently based on how fast the load is transferred to the material. The high rates of strain applied to the samples will have a much higher modulus and strength than in the actual application. The application has a cool down rate of 3 degrees per minute. It takes at least 20 minutes to achieve the operating temperature allowing the epoxy to creep and relieve some of the stresses.

Particle Physics Division
Mechanical Department Engineering Note

Herman Cease, Paul Derwent, Tom Diehl, Jim Fast, Dave Finley

May 4, 2006

Abstract

Materials testing of an adhesive for bonding Silicon to a substrate is presented. Test results include Young's Modulus, Poisson's Ratio, and the coefficient of thermal expansion at temperatures ranging from room temperature to 100 K. Data for 3 epoxies (Tra-Con F113, Epotek 301-2, Hysol 9361) are presented.

1 Introduction:

The SNAP CCD focal plane has to meet stringent performance requirements, especially on flatness. As the CCDs are manufactured at room temperature and will operate at 130 K in space, it is necessary to characterize their behavior as a function of temperature. In addition, it is expected that the CCD focal plane will undergo thermal cycles in space. As the materials (Si, AlN, SiC) have different coefficients of thermal expansion (CTE), there will be stress on the epoxy joints. NASA has criterion on the stress/strength relation for such joints [1]. This note details tensile strength tests and coefficient of thermal expansion measurements on proposed epoxies for use in the CCD assembly.

Tensile strength tests to measure Young's Modulus and Possion's Ratio were performed at Precision Measurements and Instruments Corporation (PMIC) [2] at 5 temperatures (295K, 250K, 200K, 150K, 100K). An additional measurement was made at Fermilab at 295K. Measurements of the CTE from 77 K to 295 K were also made.

Three epoxies were measured, Hysol 9361, Tra-Con F113, and Epotek 301-2. The Hysol is being considered for the AlN-SiC joint, the Tra-Con and Epotek for the CCD-AlN joint. In Table 1, we summarize the properties of the glue joints as reported by the manufacturer.

The Hysol sets in 24 hours, with a full cure in 7 days at room temperature. The epoxy samples used in the tensile strength tests had a 7 day cure. For the Hysol CTE measurements, we used a sample with a 2 day cure. For the Epotek and Tra-Con CTE measurements, measurements

Epoxy	Modulus	Viscosity	CTE @ 295 K
Hysol 9361	723 MPa	1000 Pa s	-
Tra-Con F113	-	180 cps @25 C	55 ppm/C
Epotek 301-2	-	225-425 cps	37 ppm/C

Table 1: Epoxy properties as provided by the manufacturers.

Ambient

Tra-Con F113	Elastic Modulus (psi)	356886± 12523	3.51%
	Poisson's Ratio	0.401±0.003	0.64%
	Maximum Stress (psi)	2539± 86	3.40%
Epotek 301-2	Elastic Modulus (psi)	531427± 6166	1.16%
	Poisson's Ratio	0.358±0.001	0.35%
	Maximum Stress (psi)	3751± 45	1.21%
Hysol 9361	Elastic Modulus (psi)	154678± 1526	0.99%
	Poisson's Ratio	0.433±0.007	1.67%
	Maximum Stress (psi)	1153± 9	0.77%

Table 2: Epoxy properties as measured at ambient temperatures.

250 K

Tra-Con F113	Elastic Modulus (psi)	519361± 16547	3.19%
	Poisson's Ratio	0.372 ± 0.005	1.25%
	Maximum Stress (psi)	3527± 120	3.41%
Epotek 301-2	Elastic Modulus (psi)	595903± 16547	3.19%
	Poisson's Ratio	0.365 ± 0.004	1.05%
	Maximum Stress (psi)	4115± 79	1.91%
Hysol 9361	Elastic Modulus (psi)	239242± 4375	1.83%
	Poisson's Ratio	0.435 ± 0.004	1.02%
	Maximum Stress (psi)	1736± 35	2.02%

Table 3: Epoxy properties as measured at 250 K.

were made with samples with both a 2 day cure and a 7 day cure. The Epotek epoxy has an additional manufacturing specification on residuals ions (salts) in the resin which is important for silicon bonding applications.

2 Tensile Tests

Tensile tests were performed by PMIC and also the Fermilab Material Testing Group. Both tests used samples prepared at Fermilab. The samples were dogbone shaped and machined out of cast plates of epoxy. The samples were degassed to minimize the number and shape of air bubbles. We note that the Hsyol samples did have visible bubbles on the machined surfaces.

PMIC measurements were performed per ASTM method D-638. Five dogbones of each epoxy were measured at 5 temperatures (295K, 250K, 200K, 150K, 100K). The sample modulus was calculated using the Secant Method at a 0.68% strain (or the highest strain achieved if the sample failed before that level). The Hysol samples did fail before 0.68% strain was achieved for the lower temperature measurements. Tables 2, 3, 4, 5, and 6 summarize the measurements. The full report from PMIC is included as Appendix 1.

The Fermilab Material Testing group also performed a tensile measurement at ambient temperature on the three epoxies. The steepest slope over a series of ranges was used to calculated the modulus. The crosshead pull speed was greater than 0.05 inches per minute. The results are summarized in table 7.

200 K

Tra-Con F113	Elastic Modulus (psi)	615588± 26807	4.35%
	Poisson's Ratio	0.368 ± 0.003	0.69%
	Maximum Stress (psi)	4272± 198	4.64%
Epotek 301-2	Elastic Modulus (psi)	648860± 12482	1.92%
	Poisson's Ratio	0.349 ± 0.005	1.32%
	Maximum Stress (psi)	4471± 99	2.22%
Hysol 9361	Elastic Modulus (psi)	560786± 11976	2.14%
	Poisson's Ratio	0.357 ± 0.005	1.27%
	Maximum Stress (psi)	3912± 84	2.15%

Table 4: Epoxy properties as measured at 200 K.

150 K

Tra-Con F113	Elastic Modulus (psi)	886035± 40429	4.56%
	Poisson's Ratio	0.367 ± 0.008	2.28%
	Maximum Stress (psi)	5983± 264	4.42%
Epotek 301-2	Elastic Modulus (psi)	833220± 14089	1.69%
	Poisson's Ratio	0.334 ± 0.007	2.16%
	Maximum Stress (psi)	5681± 106	1.87%
Hysol 9361	Elastic Modulus (psi)	822654± 14072	1.71%
	Poisson's Ratio	0.357 ± 0.012	3.46%
	Maximum Stress (psi)	4641± 79	1.71%

Table 5: Epoxy properties as measured at 150 K.

100 K

Tra-Con F113	Elastic Modulus (psi)	1105895± 40675	3.69%
	Poisson's Ratio	0.348 ± 0.005	1.44%
	Maximum Stress (psi)	7092± 649	9.15%
Epotek 301-2	Elastic Modulus (psi)	1014310± 14384	1.42%
	Poisson's Ratio	0.350 ± 0.008	2.34%
	Maximum Stress (psi)	6783± 162	2.39%
Hysol 9361	Elastic Modulus (psi)	1132056± 13051	1.19%
	Poisson's Ratio	0.353 ± 0.016	4.55%
	Maximum Stress (psi)	4225± 201	4.76%

Table 6: Epoxy properties as measured at 100 K.

Ambient

Tra-Con F113	Elastic Modulus (ksi)	384.84 ±85.7	22.2%
	Ultimate Tensile Strength (psi)	4798.2±569.1	11.9%
Epotek 301-2	Elastic Modulus (ksi)	650.6± 176.2	% 27.1
	Ultimate Tensile Strength (psi)	9664.2±1769.6	18.3%
Hysol 9361	Elastic Modulus (ksi)	137.5± 17	12.3%
	Ultimate Tensile Strength (psi)	2400.4± 190.6	7.94%

Table 7: Epoxy properties as measured at ambient temperatures by the Fermilab Material Properties Testing group.

3 Coefficient of Thermal Expansion Measurements

CTE measurements were made at Fermilab. The measurements were performed in the spirit of ASTM-E831. Samples were approximately 8 mm × 8 mm × 20 mm, machined from samples cast in a mold. Each sample was vacuum degassed during the casting to minimize the size and number of trapped gas bubbles.

The CTE was measured by placing the sample in a holder inside of a cryostat. Liquid nitrogen is poured into the cryostat. Once the temperature stabilized at 77 K, a heater is used to ramp the temperature to ambient temperature with a rate of 1-2deg C/minute. An LVDT at the top of the sample measured the change in sample length. The length and temperature were recorded during the cooldown and the warmup. In figure 1, we show a picture of the sample in the holder. The LVDT is at the top of the picture, connected via quartz rods to the sample holder. The sample holder is installed inside the cryostat. We report the integral fractional change in length (dL/L) of the sample as a function of temperature in table 8. In figures ??, ??, and ??, we show the fractional change in length as a function of temperature for one sample of Tra-Con F113, Epotek 301-2, and Hysol 9361. In figure ??, we show a representative time ramp for one of the measurements.

4 Stress/Strength Ratios

The NASA guideline for epoxy joints [1] is a safety margin of a factor of 2 on the stress. With the measured modulus and CTE, we can calculate the expected stress on the joint and compare to yield strength, using the following logic:

$$\begin{aligned} \text{Modulus} &= \frac{\text{Stress}}{\text{Strain}} \\ \text{Strain} &= \frac{\text{dL}}{\text{L}} \\ \frac{\text{dL}}{\text{L}}\,(\text{epoxy}) &= \text{CTE (epoxy)} \times \Delta\text{T} \end{aligned} \tag{1}$$

With the assumption that the CTE of the substrate is small compared to the epoxy, the stress on the joint is simply:

$$\text{Stress} = \text{Modulus} \times \text{CTE (epoxy)} \times \Delta\text{T} \tag{2}$$

A criterion that the stress must be less than $\frac{1}{2}$ the yield strength of the epoxy leads to the requirement that

$$\frac{\text{Modulus} \times \text{CTE (epoxy)} \times \Delta\text{T}}{\text{yield strength (epoxy)}} < \frac{1}{2}. \tag{3}$$

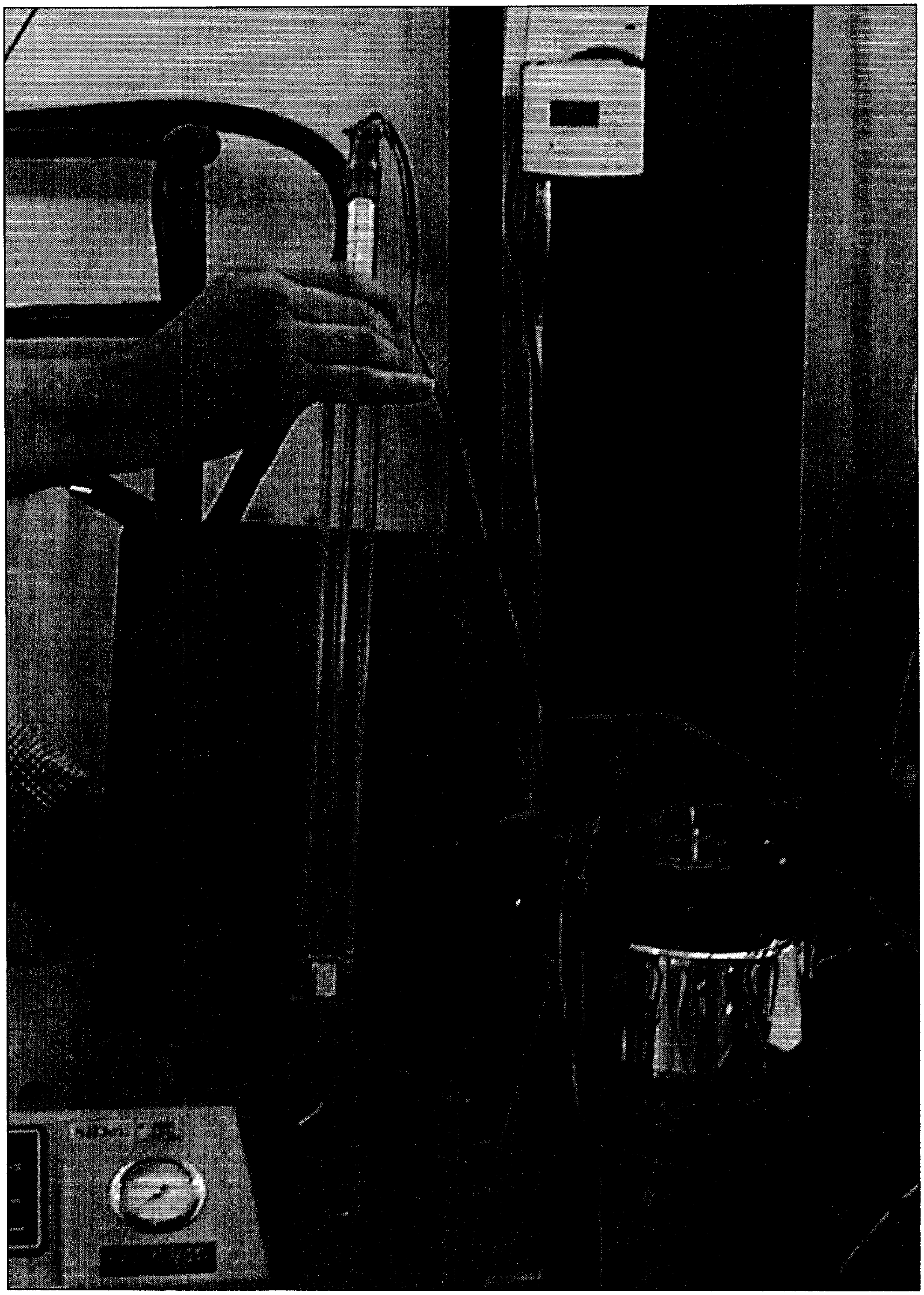

Figure 1: The sample holder for use in the CTE measurements.

Ambient		
Tra-Con F113 dL/L ($\times 10^{-3}$)	–	–
Epotek 301-2 dL/L ($\times 10^{-3}$)	–	–
Hysol 9361 dL/L ($\times 10^{-3}$)	–	–
250 K		
Tra-Con F113 dL/L ($\times 10^{-3}$)	-3.27 $\pm$ 0.13	3.94%
Epotek 301-2 dL/L ($\times 10^{-3}$)	-2.83 $\pm$ 0.04	1.52%
Hysol 9361 dL/L ($\times 10^{-3}$)	-4.69 $\pm$ 0.04	0.8%
200 K		
Tra-Con F113 dL/L ($\times 10^{-3}$)	-6.20 $\pm$ 0.11	1.73%
Epotek 301-2 dL/L ($\times 10^{-3}$)	-5.45 $\pm$ 0.03	0.59%
Hysol 9361 dL/L ($\times 10^{-3}$)	-8.69 $\pm$ 0.02	0.3%
150 K		
Tra-Con F113 dL/L ($\times 10^{-3}$)	-8.71 $\pm$ 0.06	0.76%
Epotek 301-2 dL/L ($\times 10^{-3}$)	-7.70 $\pm$ 0.11	1.37%
Hysol 9361 dL/L ($\times 10^{-3}$)	-11.3 $\pm$ 0.003	0.2%
100 K		
Tra-Con F113 dL/L ($\times 10^{-3}$)	-10.7 $\pm$ 0.08	0.72%
Epotek 301-2 dL/L ($\times 10^{-3}$)	-9.66 $\pm$ 0.18	1.88%
Hysol 9361 dL/L ($\times 10^{-3}$)	-11.3 $\pm$ 0.03	0.2%

Table 8: CTE for the three epoxies as measured at the 5 temperatures.

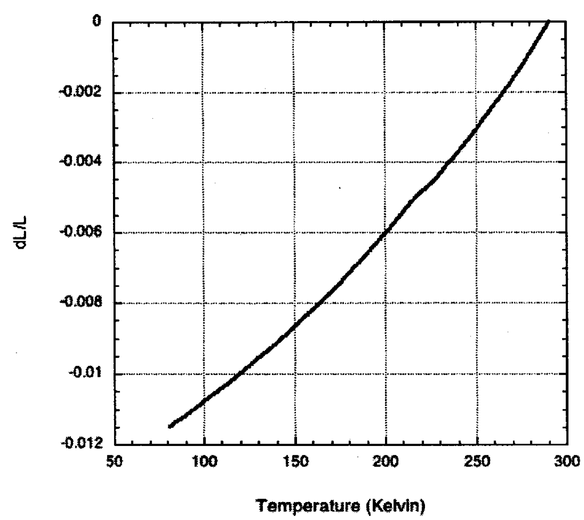

Figure 2: The integral dL/L vs temperature for a representative Tra-Con F113 sample.

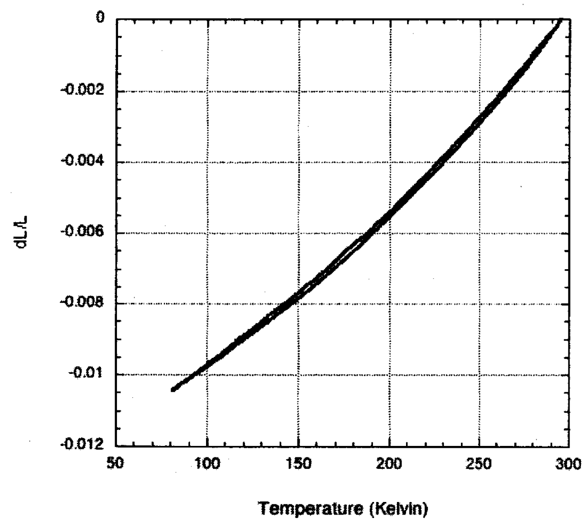

Figure 3: The integral dL/L vs temperature for a representative Epotek 301-2 sample.

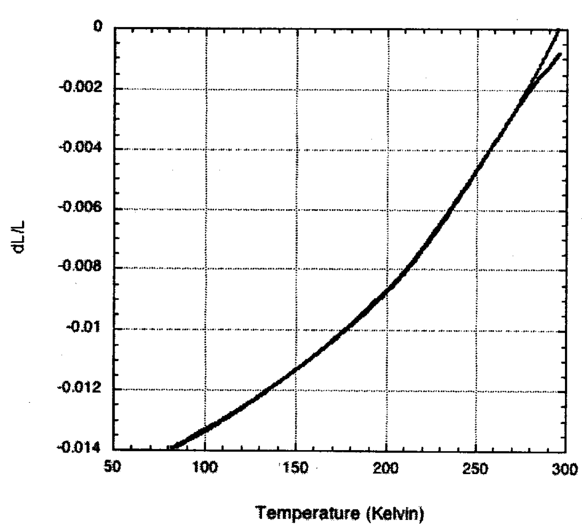

Figure 4: The integral dL/L vs temperature for a representative Hysol 9361 sample.

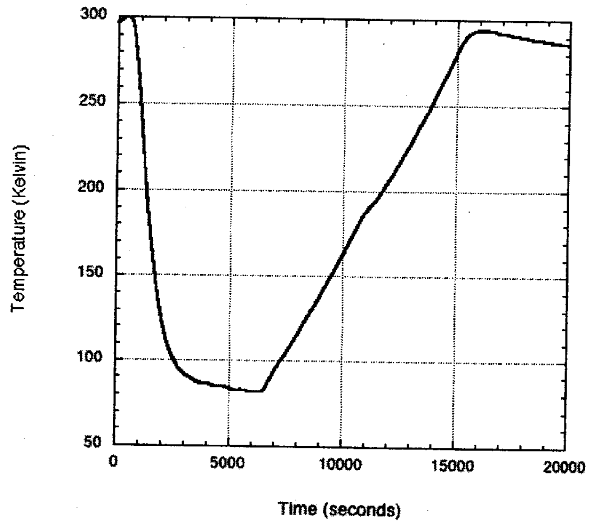

Figure 5: The temperature vs time for a representative CTE measurement.

	Ambient	250 K	200 K	150 K	100 K
Tra-Con F113	–	0.36	0.89	1.32	1.55
Epotek 301-2	–	0.17	0.37	0.67	1.0
Hysol 9361	–	0.47	1.2	2.0	3.5

Table 9: The ratio of stress to strain as defined in equation 3.

The stress reported in the data sets is not the epoxy yield strength. However, a conservative guideline can still be determined if the maximum stress achieved is used in the calculations as a proof stress. We will use either the room temperature ultimate strength or the maximum stress applied to the sample at temperature, whichever is larger. As the strength is known to increase with decreasing temperature, this selection is a conservative approach. In Table 9, we show the ratio of stress over strength as defined above.

The Epotek has the lowest bond stress and, at ambient temperature, the highest ratio of maximum strength to strength at 0.68%. Although none of the epoxies meet the criteria for temperatures below 200 K, it does not mean the joint will fail. We have chosen to take a conservative approach in the calculation of the maximum stress. In addition, the tensile strength data collected was taken at high pull rates (0.10 inch/minute for the PMIC tests). Epoxies, as with most plastics, are visco-elastic materials which respond differently based on how quickly the load is transferred to the material. The high rates of strain applied to the samples during testing will have a much higher modulus and stress than in the actual application. During flight, the CCD focal plane will have a cool down rate of 3 degrees per minute, taking at least 20 minutes to achieve operating temperature and allowing the epoxy to creep and relieve some of the stresses.

5 Conclusions

We have presented measurements on epoxy properties for use in the SNAP CCD assembly, covering temperatures from ambient to 100 K. Test results on Young's Modulus, Poisson's Ratio, and CTE have been presented. A criterion for the epoxy has been proposed.

References

[1] NASA-STD-5001 - Structural Design and Test Factors of Safety for Spaceflight Hardware

[2] Precision Measurements and Instruments Corporation, 3665 SW Deschutes Street, Corvallis, OR 97333

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PURCHASE ORDER NUMBER 565015

ELASTIC PROPERTY MEASUREMENTS OF EPOXY SPECIMENS

February 28, 2006

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ELASTIC PROPERTY MEASUREMENTS OF EPOXY SPECIMENS

WORK CONDUCTED FOR FERMILAB PURCHASE ORDER NUMBER 565015

February 28, 2006

Precision Measurements and Instruments Corporation measured the elastic properties of 75 epoxy resin specimens. Coupons were prepared for Modulus testing and Poisson's Ratio testing. Testing was conducted at 100K, 150K, 200K, 250K and ambient temperatures. Testing was performed per ASTM method D-638. Results are presented in the attached tables. A brief description of the test procedure, data analysis and comments on the results follow.

Specimen Description

Fermilab provided the following specimens:

Quantity	Description	Length	Width	Thickness
35	Tracon F113	8.0"	1.0"	0.125"
35	Epo-Tek 301-2	8.0"	1.0"	0.125"
33	Hysol ES 9361	8.0"	1.0"	0.125"

Test Procedure

♦ Specimen Check-In

The specimens were received on December 19th, January 11th and January 12th, via Federal Express. The specimens were inspected for damage. No damage was observed. However, it was noted that the Hysol specimens had bubbles visible on the machined surfaces. The specimens were stored at room temperature prior to measurement. A complete list of the specimens is located at the end of the report.

♦ Specimen Preparation

The specimen preparation and strain gage attachment procedures suggested by the strain gage supplier were followed. Micro-Measurements CEA-06-125UT-350 strain gages were used for both axial and lateral strain measurement on the specimens. Strain gages were bonded to each side of the specimen at corresponding positions, using Micro-Measurements M-Bond AE-10 adhesive. The specimens were clamped and cured for 6+ hours at ≤95°F. Each gage was then wired in series to the corresponding gage on the opposite surface to account for specimen bending. In a few cases the strain gages were shifted axially on the specimen to avoid placing them close to the bubbles.

♦ Test Procedure

The tensile test machine crosshead speed was set to 0.10 inch/minute. 2 VDC strain gage excitation was used. The specimens were first secured in the machine with the top wedge grip, centered and aligned with the direction of force. For the low temperature tests thermocouples were placed at three points in the vicinity of the strain gages. The gauge portion of the specimen was enclosed in an insulated chamber. Liquid nitrogen was used to achieve cooling for the 100 K, 150 K, 200 K and 250 K tests. The cryogen was directed close to the proximity of the specimens on either side by dual spray bars. Approximately 20-60 minutes was allowed for the specimens to equilibrate at the desired temperatures. At this point the strain gage conditioning circuitry was balanced, the lower wedge grip tightened and the test started. The load was applied to the specimens by movement of the upper grip until the limits of the strain gage were reached, failure occurred, or the specimens began to yield outside of the gauge section. Load, strain and temperature data were recorded every half second.

- Analysis

The various elastic properties were calculated over certain strain ranges selected to coincide with a level obtained by all, or nearly all, specimens. The first data point used for all calculations was generally the first positive point after all slack was removed. The last data point was generally the limit of the axial strain gage/electronics, ~6800 μ-strain. In some cases the last point was limited by breakage or yielding outside of the temperature controlled zone. In a few cases the endpoints were shifted somewhat due to unusual temperature fluctuations. The secant modulus was calculated by taking the change in axial stress divided by the change in axial strain between the chosen endpoints. Poisson's Ratio was calculated by using the change in transverse strain divided by the change in axial strain at the same endpoints. The nominal dimensions were used in all of the calculations.

Test Results

The results are presented in tabular format in **Table 1**. The stress and strain of the maximum endpoint at which each property is calculated are listed with the results. The properties may be determined for other strain ranges. The raw stress and strain data is being supplied to the requesting engineer.

Some of the specimens broke during the test. The location of the break was always in the area of the specimen held by the upper grip or right beneath it. All but two of the broken specimens were in the Hysol group. In this group, three out of five of the 100 K specimens broke, all of the 150 K specimens broke, and one of the ambient specimens broke. This could have been due to bubbles which were visible in these specimens. In every case of a break, a bubble could be seen in the fractured surfaces. In one of the Hysol specimens the strain gage was slightly offset vertically in order to avoid a bubble. This specimen did not break. In addition, one of the Epo-Tek specimens broke, at 200 K, and one of the Tracon specimens broke, at 100 K.

Possible sources of error include the presence of bubbles in the specimens, the impact of the strain gages on the material and temperature gradients. Digital control of the cryogen flow also induced some degree of temperature fluctuation, which is evident in some of the strain data.

Please contact our technical staff at (541) 753-0607 if you have any questions or require additional information regarding these measurements.

Submitted by:

Don Schneider	Ann Gaidos-Morgan
Project Engineer	Test Technician

Precision Measurements and Instruments Corporation hereby claims that test results are obtained by techniques based on relevant ASTM standards, calibrations with NIST standard reference materials and/or published procedures. Thus, we accept no liability for test results beyond the cost of the contract rendered.

TABLE 1, ELASTIC MODULUS AND POISSON'S RATIO

Ambient (295 K)				
Specimen ID	Elastic Modulus psi	Poisson's Ratio	Maximum Strain %	Maximum Stress psi
TB_5_5	327,203	0.394	0.68	2,324
TB_6_5	369897	0.407	0.69	2,637
TB_8_5	390047	0.405	0.69	2,771
TB_9_5	369262	0.403	0.68	2,611
TB_11_5	328019	0.396	0.69	2,353
ave	**356,886**	**0.401**		2539
st dev	**28,002**	**0.006**		
ET_1_5	541511	0.353	0.69	3,808
ET_2_5	537859	0.358	0.69	3,791
ET_7_5	542696	0.360	0.69	3,852
ET_19_5	510312	0.359	0.69	3,596
ET_20_5	524758	0.358	0.69	3,708
ave	**531,427**	**0.358**		~~[illegible]~~
st dev	**13,787**	**0.003**		3751
HS_14_5	155524	0.452	0.69	1,158
HS_15_5	159339	0.434	0.68	1,175
HS_16_5	150281	0.408	0.68	1,122
HS_17_5	155573	0.433	0.68	1,162
HS_18_5	152674	0.439	0.69	1,149
ave	**154,678**	**0.433**		1153
st dev	**3,413**	**0.016**		

250 K				
Specimen ID	Elastic Modulus psi	Poisson's Ratio	Maximum Strain %	Maximum Stress psi
TB_5_4	513,054	0.369	0.67	3,479
TB_6_4	522,770	0.356	0.68	3,591
TB_8_4	545,348	0.373	0.67	3,463
TB_9_4	555,162	0.383	0.69	3,924
TB_11_4	460,474	0.378	0.67	3,179
ave	519,361	0.372		3527
st dev	37,000	0.010		
ET_1_4	637,210	0.365	0.67	4,355
ET_2_4	571,547	0.364	0.69	4,013
ET_7_4	604,671	0.359	0.69	4,239
ET_19_4	568,324	0.357	0.68	3,973
ET_20_4	597,763	0.379	0.69	4,046
ave	595,903	0.365		4125
st dev	28,020	0.009		
HS_14_4	233,015	0.442	0.68	1,669
HS_15_4	225,696	0.425	0.69	1,644
HS_17_4	249,646	0.446	0.69	1,812
HS_18_4	245,975	0.438	0.70	1,813
HS_16_6	241,877	0.424	0.68	1,744
ave	239,242	0.435		1736
st dev	9,782	0.010		

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TABLE 1, ELASTIC MODULUS AND POISSON'S RATIO

200 K				
Specimen ID	Elastic Modulus psi	Poisson's Ratio	Maximum Strain %	Maximum Stress psi
TB_5_3	607,451	0.367	0.69	4258
TB_6_3	613,755	0.370	0.68	4295
TB_8_3	651,835	0.371	0.68	4559
TB_11_3	522,938	0.359	0.68	3550
TB_9_6	681,959	0.374	0.67	4698
ave	**615,588**	**0.368**		4272
st dev	**59,943**	**0.006**		
ET_1_3	634,469	0.344	0.68	4421
ET_7_3	678,932	0.358	0.68	4738
ET_19_3	608,731	0.333	0.68	4181
ET_20_3	654,185	0.351	0.68	4374
ET_2_3+	667,985	0.357	0.68	4642
ave	**648,860**	**0.349**		4471
st dev	**27,910**	**0.010**		
HS_14_3	603,769	0.347	0.69	4229
HS_15_3	536,650	0.351	0.69	3734
HS_16_3	561,845	0.366	0.69	3901
HS_17_3	561,774	0.369	0.68	3870
HS_18_3	539,892	0.351	0.69	3825
ave	**560,786**	**0.357**		3912
st dev	**26,780**	**0.010**		

150 K				
Specimen ID	Elastic Modulus psi	Poisson's Ratio	Maximum Strain %	Maximum Stress psi
TB_5_2	873,958	0.355	0.68	5880
TB_6_2	911,669	0.365	0.69	6262
TB_8_2	939,767	0.358	0.69	6384
TB_9_2	968,010	0.399	0.68	6393
TB_11_2	736,770	0.356	0.69	4994
ave	**886,035**	**0.367**		5982
st dev	**90,402**	**0.019**		
ET_1_2	825,968	0.327	0.68	5364
ET_2_2	869,247	0.351	0.68	5795
ET_7_2	852,639	0.319	0.69	5932
ET_19_2	785,872	0.322	0.69	5501
ET_20_2	832,372	0.352	0.69	5814
ave	**833,220**	**0.334**		5681
st dev	**31,505**	**0.016**		
HS_14_2	781,235	0.322	0.59	4616
HS_15_2	858,394	0.365	0.54	4596
HS_16_2	817,672	0.394	0.59	4885
HS_18_2	849,032	0.365	0.54	4709
HS_17_6	806,935	0.338	0.55	4398
ave	**822,654**	**0.357**		4641
st dev	**31,467**	**0.028**		

TABLE 1, ELASTIC MODULUS AND POISSON'S RATIO

100 K				
Specimen ID	Elastic Modulus psi	Poisson's Ratio	Maximum Strain %	Maximum Stress psi
TB_5_1	1,115,620	0.361	0.69	7518
TB_6_1	1,084,636	0.351	0.69	7671
TB_8_1	1,176,787	0.331	0.69	7982
TB_9_1	1,190,017	0.352	0.69	7777
TB_11_1	962,414	0.344	0.45	4514
ave	**1,105,895**	**0.348**		7737
st dev	**91,153**	**0.011**		
ET_1_1	1,002,227	0.359	0.69	6360
ET_2_1	1,022,622	0.326	0.69	6570
ET_7_1	1,062,459	0.375	0.69	7327
ET_20_1	1,009,726	0.347	0.69	6865
ET_19_1	974,514	0.343	0.69	6794
ave	**1,014,310**	**0.350**		6783
st dev	**32,164**	**0.018**		
HS_14_1	1,095,470	0.367	0.39	4223
HS_15_1	1,121,990	0.314	0.39	4479
HS_16_1	1,177,256	0.317	0.41	4789
HS_17_1	1,141,779	0.394	0.31	3602
HS_18_1	1,123,786	0.374	0.40	4033
ave	**1,132,056**	**0.353**		4225
st dev	**30,190**	**0.036**		

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The following table is a listing of the Tracon test specimens.

1) Tracon F113 TB_5_1 (100K) 8" X 1" X 0.13"	18) Tracon F113 TB_8_4 (250K) 8" X 1" X 0.13"
2) Tracon F113 TB_5_2 (150K) 8" X 1" X 0.13"	19) Tracon F113 TB_8_5 (300K) 8" X 1" X 0.13"
3) Tracon F113 TB_5_3 (200K) 8" X 1" X 0.13"	20) Tracon F113 TB_9_1 (100K) 8" X 1" X 0.13"
4) Tracon F113 TB_5_4 (250K) 8" X 1" X 0.13"	21) Tracon F113 TB_9_2 (150K) 8" X 1" X 0.13"
5) Tracon F113 TB_5_5 (300K) 8" X 1" X 0.13"	22) Tracon F113 TB_9_3 (200K) 8" X 1" X 0.13"
6) Tracon F113 TB_5_6 (Spare) 8" X 1" X 0.13"	23) Tracon F113 TB_9_4 (250K) 8" X 1" X 0.13"
7) Tracon F113 TB_5_7 (Spare) 8" X 1" X 0.13"	24) Tracon F113 TB_9_5 (300K) 8" X 1" X 0.13"
8) Tracon F113 TB_6_1 (100K) 8" X 1" X 0.13"	25) Tracon F113 TB_9_6 (Spare) 8" X 1" X 0.13"
9) Tracon F113 TB_6_2 (150K) 8" X 1" X 0.13"	26) Tracon F113 TB_11_1 (100K) 8" X 1" X 0.13"
10) Tracon F113 TB_6_3 (200K) 8" X 1" X 0.13"	27) Tracon F113 TB_11_2 (150K) 8" X 1" X 0.13"
11) Tracon F113 TB_6_4 (250K) 8" X 1" X 0.13"	28) Tracon F113 TB_11_3 (200K) 8" X 1" X 0.13"
12) Tracon F113 TB_6_5 (300K) 8" X 1" X 0.13"	29) Tracon F113 TB_11_4 (250K) 8" X 1" X 0.13"
13) Tracon F113 TB_6_6 (Spare) 8" X 1" X 0.13"	30) Tracon F113 TB_11_5 (300K) 8" X 1" X 0.13"
14) Tracon F113 TB_6_7 (Spare) 8" X 1" X 0.13"	31) Tracon F113 TB_11_6 (Spare) 8" X 1" X 0.13"
15) Tracon F113 TB_8_1 (100K) 8" X 1" X 0.13"	32) Tracon F113 TB_12_1 (Spare) 8" X 1" X 0.13"
16) Tracon F113 TB_8_2 (150K) 8" X 1" X 0.13"	33) Tracon F113 TB_12_2 (Spare) 8" X 1" X 0.13"
17) Tracon F113 TB_8_3 (200K) 8" X 1" X 0.13"	34) Tracon F113 TB_12_3 (Spare) 8" X 1" X 0.13"
	35) Tracon F113 TB_12_4 (Spare) 8" X 1" X 0.13"

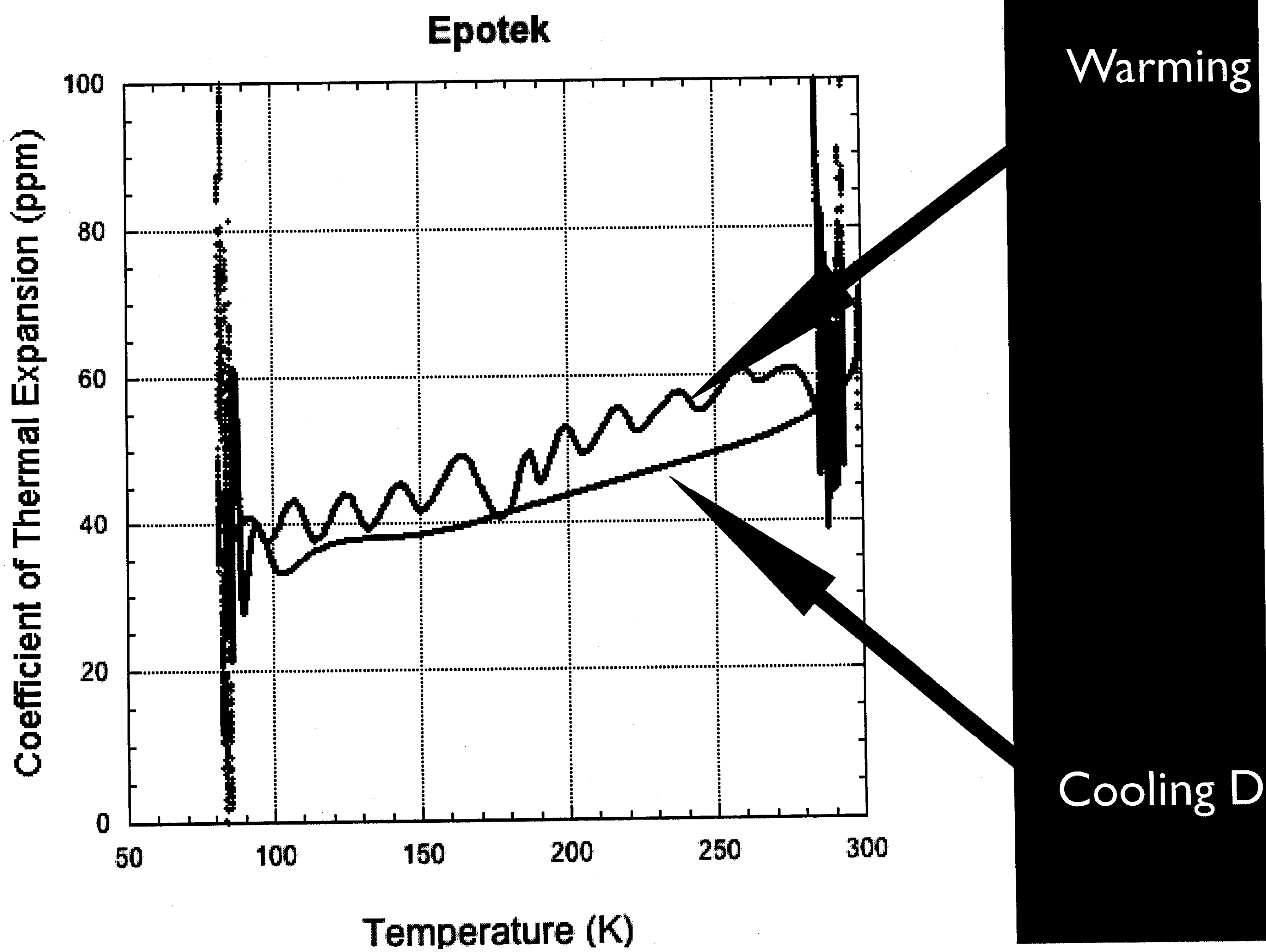
The following table is a listing of the Epo-Tek 301-2 specimens.

36) Epo-Tek 301-2 ET_1_1 (100K) 8" X 1" X 0.13"	53) Epo-Tek 301-2 ET_4_2 (Spare) 8" X 1" X 0.13"
37) Epo-Tek 301-2 ET_1_2 (150K) 8" X 1" X 0.13"	54) Epo-Tek 301-2 ET_7_1 (100K) 8" X 1" X 0.13"
38) Epo-Tek 301-2 ET_1_3 (200K) 8" X 1" X 0.13"	55) Epo-Tek 301-2 ET_7_2 (150K) 8" X 1" X 0.13"
39) Epo-Tek 301-2 ET_1_4 (250K) 8" X 1" X 0.13"	56) Epo-Tek 301-2 ET_7_3 (200K) 8" X 1" X 0.13"
40) Epo-Tek 301-2 ET_1_5 (300K) 8" X 1" X 0.13"	57) Epo-Tek 301-2 ET_7_4 (250K) 8" X 1" X 0.13"
41) Epo-Tek 301-2 ET_2_1 (100K) 8" X 1" X 0.13"	58) Epo-Tek 301-2 ET_7_5 (300K) 8" X 1" X 0.13"
42) Epo-Tek 301-2 ET_2_2 (150K) 8" X 1" X 0.13"	59) Epo-Tek 301-2 ET_19_1 (100K) 8" X 1" X 0.13"
43) Epo-Tek 301-2 ET_2_3 (200K) 8" X 1" X 0.13"	60) Epo-Tek 301-2 ET_19_2 (150K) 8" X 1" X 0.13"
44) Epo-Tek 301-2 ET_2_4 (250K) 8" X 1" X 0.13"	61) Epo-Tek 301-2 ET_19_3 (200K) 8" X 1" X 0.13"
45) Epo-Tek 301-2 ET_2_5 (300K) 8" X 1" X 0.13"	62) Epo-Tek 301-2 ET_19_4 (250K) 8" X 1" X 0.13"
46) Epo-Tek 301-2 ET_2_6(Spare) 8" X 1" X 0.13"	63) Epo-Tek 301-2 ET_19_5 (300K) 8" X 1" X 0.13"
47) Epo-Tek 301-2 ET_3_1 (Spare) 8" X 1" X 0.13"	64) Epo-Tek 301-2 ET_19_6 (Spare) 8" X 1" X 0.13"
48) Epo-Tek 301-2 ET_3_2 (Spare) 8" X 1" X 0.13"	65) Epo-Tek 301-2 ET_20_1 (100K) 8" X 1" X 0.13"
49) Epo-Tek 301-2 ET_3_3 (Spare) 8" X 1" X 0.13"	66) Epo-Tek 301-2 ET_20_2 (150K) 8" X 1" X 0.13"
50) Epo-Tek 301-2 ET_3_4 (Spare) 8" X 1" X 0.13"	67) Epo-Tek 301-2 ET_20_3 (200K) 8" X 1" X 0.13"
51) Epo-Tek 301-2 ET_3_5 (Spare) 8" X 1" X 0.13"	68) Epo-Tek 301-2 ET_20_4 (250K) 8" X 1" X 0.13"
52) Epo-Tek 301-2 ET_4_1 (Spare) 8" X 1" X 0.13"	69) Epo-Tek 301-2 ET_20_5 (300K) 8" X 1" X 0.13"
	70) Epo-Tek 301-2 ET_20_6 (Spare) 8" X 1" X 0.13"

The following table is a listing of the HYSOL 9361 HS Specimens.

71) HYSOL 9361 HS_14_1 (100K) 8" X 1" X 0.13"	89) HYSOL 9361 HS_16_6 (Spare) 8" X 1" X 0.13"
72) HYSOL 9361 HS_14_2 (150K) 8" X 1" X 0.13"	90) HYSOL 9361 HS_16_7 (Spare) 8" X 1" X 0.13"
73) HYSOL 9361 HS_14_3 (200K) 8" X 1" X 0.13"	91) HYSOL 9361 HS_17_1 (100K) 8" X 1" X 0.13"
74) HYSOL 9361 HS_14_4 (250K) 8" X 1" X 0.13"	92) HYSOL 9361 HS_17_2 (150K) 8" X 1" X 0.13"
75) HYSOL 9361 HS_14_5 (300K) 8" X 1" X 0.13"	93) HYSOL 9361 HS_17_3 (200K) 8" X 1" X 0.13"
76) HYSOL 9361 HS_14_6 (Spare) 8" X 1" X 0.13"	94) HYSOL 9361 HS_17_4 (250K) 8" X 1" X 0.13"
77) HYSOL 9361 HS_15_1 (100K) 8" X 1" X 0.13"	95) HYSOL 9361 HS_17_5 (300K) 8" X 1" X 0.13"
78) HYSOL 9361 HS_15_2 (150K) 8" X 1" X 0.13"	96) HYSOL 9361 HS_17_6 (Spare) 8" X 1" X 0.13"
79) HYSOL 9361 HS_15_3 (200K) 8" X 1" X 0.13"	97) HYSOL 9361 HS_18_1 (100K) 8" X 1" X 0.13"
80) HYSOL 9361 HS_15_4 (250K) 8" X 1" X 0.13"	98) HYSOL 9361 HS_18_2 (150K) 8" X 1" X 0.13"
81) HYSOL 9361 HS_15_5(300K) 8" X 1" X 0.13"	99) HYSOL 9361 HS_18_3 (200K) 8" X 1" X 0.13"
82) HYSOL 9361 HS_15_6 (Spare) 8" X 1" X 0.13"	100) HYSOL 9361 HS_18_4 (250K) 8" X 1" X 0.13"
83) HYSOL 9361 HS_15_7 (Spare) 8" X 1" X 0.13"	101) HYSOL 9361 HS_18_5 (300K) 8" X 1" X 0.13"
84) HYSOL 9361 HS_16_1 (100K) 8" X 1" X 0.13"	102) HYSOL 9361 HS_18_6 (Spare) 8" X 1" X 0.13"
85) HYSOL 9361 HS_16_2 (150K) 8" X 1" X 0.13"	103) HYSOL 9361 HS_18_7 (Spare) 8" X 1" X 0.13"
86) HYSOL 9361 HS_16_3 (200K) 8" X 1" X 0.13"	
87) HYSOL 9361 HS_16_4 (250K) 8" X 1" X 0.13"	
88) HYSOL 9361 HS_16_5 (300K) 8" X 1" X 0.13"	

See hysteresis in the cool down, warm up cycle!



Dump LN_2 in dewar

allow sample to cool

Ramp up to 295 K over 3 hour time scale

- 'spirit of ASTM-E831'
- 10 W heater
- temperature control feedback different than sample temperature measure